



ICSSP5: BHUBANESWAR (2012)

Primary-Dendrite Array: Observations from Ground-based and Space Station Processed Samples

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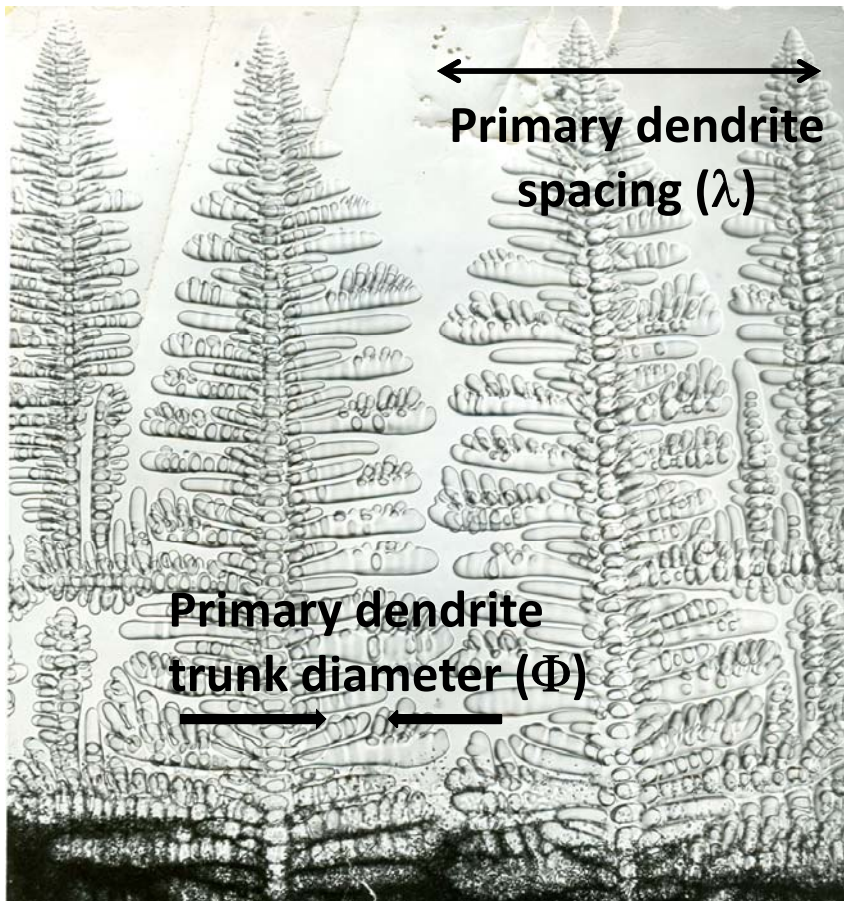
Collaborative Effort between NASA and European Space Agency : Program MICAST

(Microstructure Formation in Castings of Technical Alloys under Diffusive and Magnetically Controlled Convective Conditions)

Introduction

- Why “low gravity” DS
- MICAST6 and MICAST 7 processing details
- Microstructure along DS length
- Primary dendrite spacing
- Primary dendrite trunk diameter
- Surprises

Dendritic array morphology depends upon DS processing parameters: G , R , C_o (convection?)



1. *Primary dendrite arm spacing (λ):*
Extensive literature (SCN/Metals)
2. Secondary/tertiary arm spacing:
Extensive-SCN/Metals
3. Dendrite tip radius: SCN/ limited (Al-Cu, Pb-Au, Pb-Pd)
4. *Primary dendrite trunk diameter (Φ):*
Limited (Esaka:Thesis-86, Grugel: 92/95)

Natural convection is inevitable during DS on earth

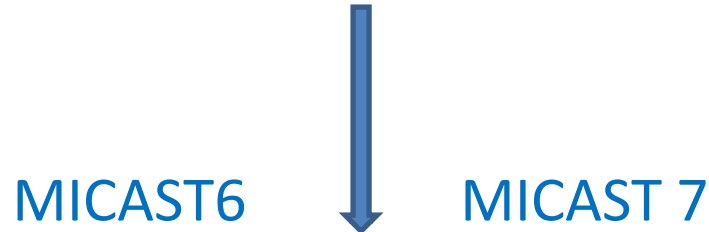
Thermally stable

- *Solutally stable (Hypoeutectic Al-Cu)*
“dendrite steeping” (radial macrosegregation)
- *Solutally unstable (Hypoeutectic Pb-Sn)*
“axial macrosegregation and freckle”

Dendritic array growth under diffusive solutal and thermal transport only possible in the absence of “g”

Purpose

MICAST: A systematic analysis of the effect of convection on the microstructural evolution in cast binary, ternary and commercial Al-Si based alloys.



Re-melt and DS terrestrially grown dendritic mono-crystals of Al-7 wt% Si (9-mm dia, 25 cm long) in μg .

Advantages:	Minimize Thermo-Solutal Convection
Intent:	Produce Segregation Free Samples Grown Under Diffusion-Controlled Conditions
Purpose:	Better Understand the Relationship between Processing and Microstructure-Development

Microgravity Processing : Partially remelt and then DS from terrestrially grown dendritic mono-crystal in μg .



(Al-7%Si Single Crystal Dendritic)



ESA- Sample Cartridge Assembly

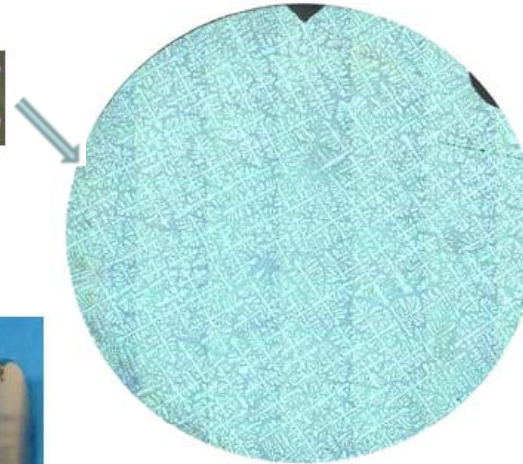


ESA_MSL Low Gradient Furnace



ESA:
Material
Science
Laboratory

NASA_MSSR-1 Flight Rack



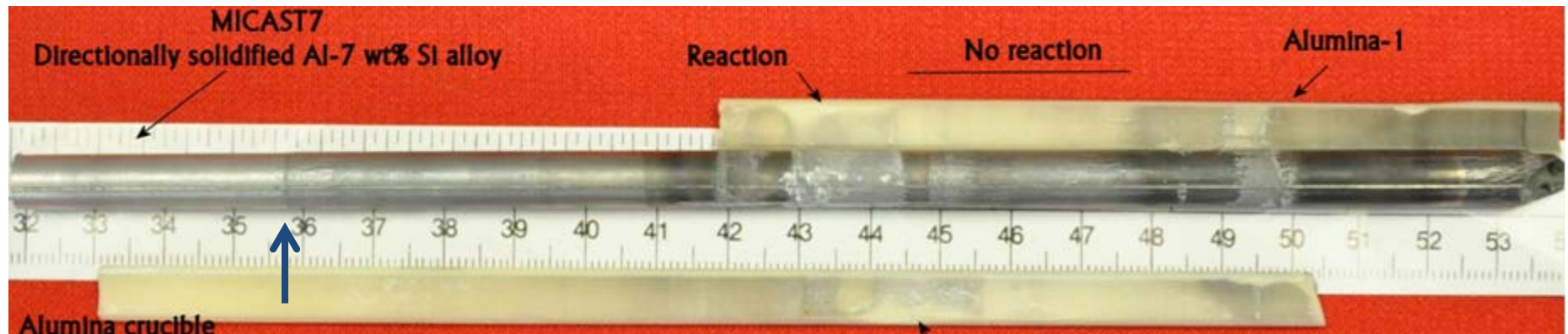
Transverse View

Microgravity Processed Sample MICAST 7



Eutectic Melt Back
/ Isotherm

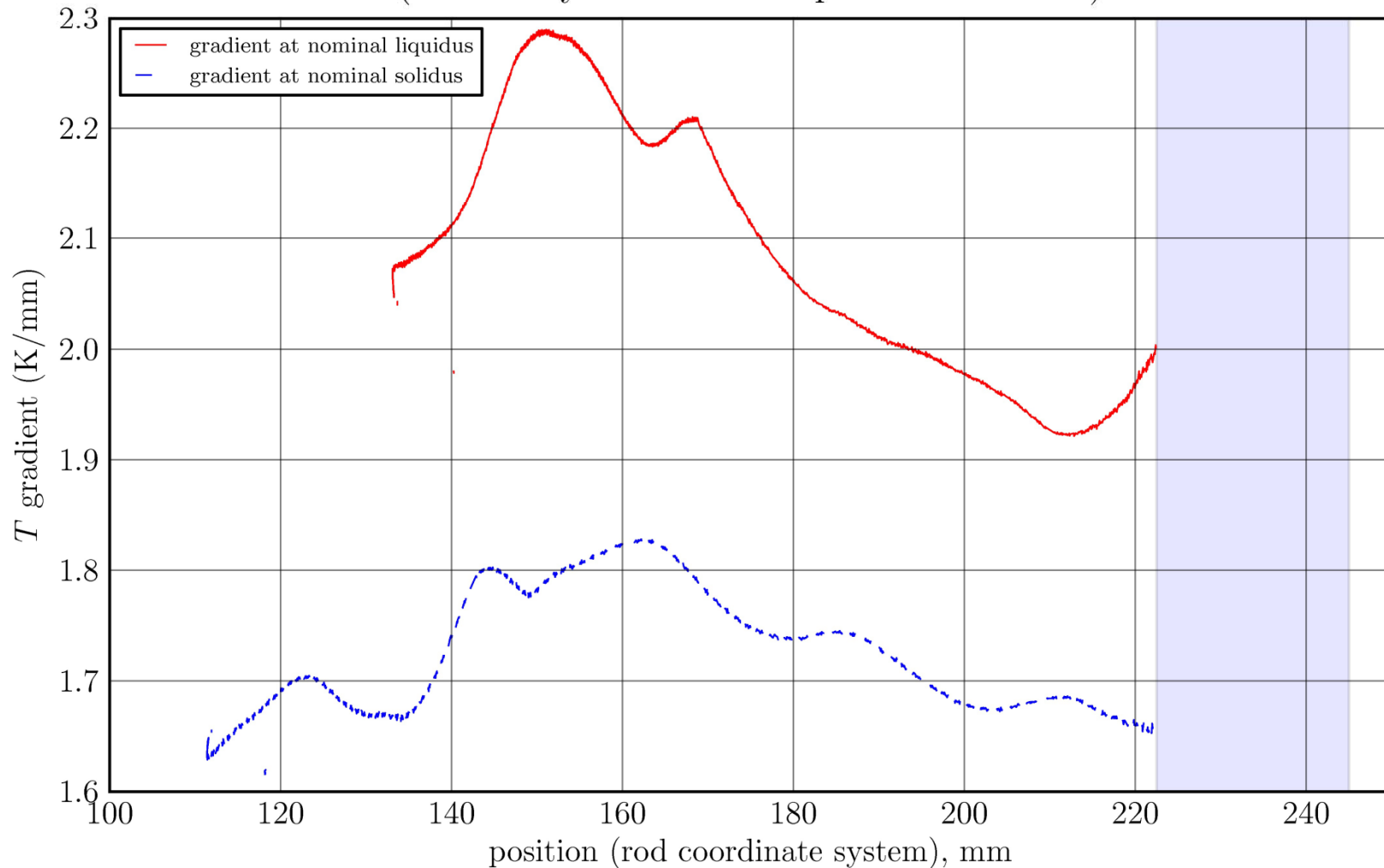
X-ray radiograph of MICAST7



Eutectic Melt Back

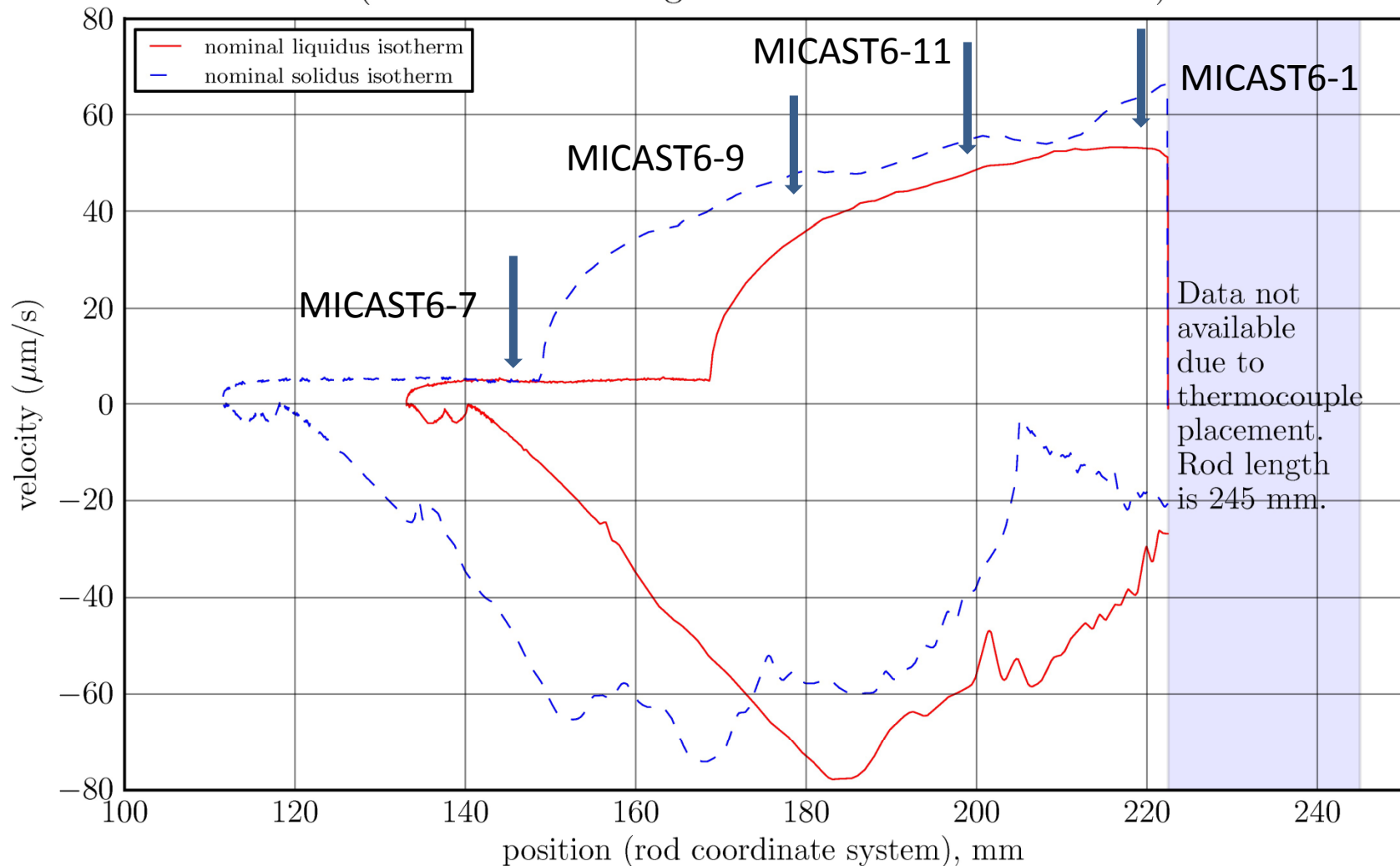
MICAST6: ESA-Low Gradient Furnace (1-hr heat-up, 5-hr hold, $G_l \sim 20 \text{ K cm}^{-1}$): 3.8 cm at $5 \mu\text{m s}^{-1}$, 11.3 cm at $50 \mu\text{m s}^{-1}$

temperature gradients at nominal liquidus and solidus isotherms
(note: only solidification portion is shown)



MICAST6: ESA-Low Gradient Furnace (1-hr heat-up, 5-hr hold, $G_1 \sim 20 \text{ K cm}^{-1}$): 3.8 cm at $5 \mu\text{m s}^{-1}$, 11.3 cm at $50 \mu\text{m s}^{-1}$

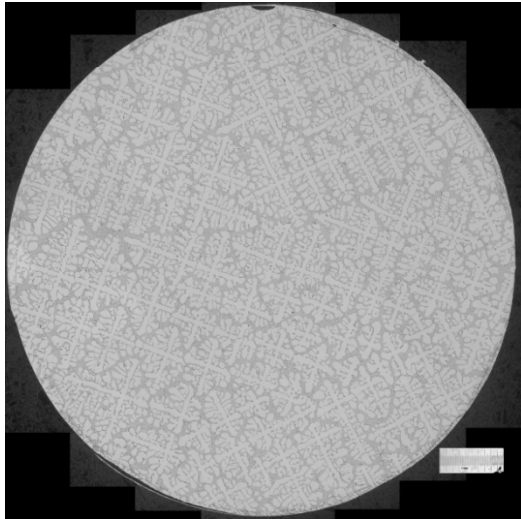
isotherm velocity vs. position along the Al-Si rod
(note: both melting and solidification are shown)



Comparison of microstructures: Al-7% Si directionally solidified on ground and on ISS (MICAST6)

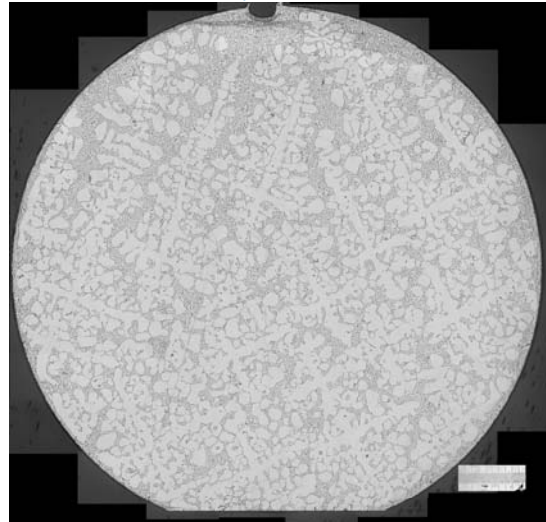
MICAST6 SEED

41 K cm^{-1} , $22 \mu\text{m s}^{-1}$

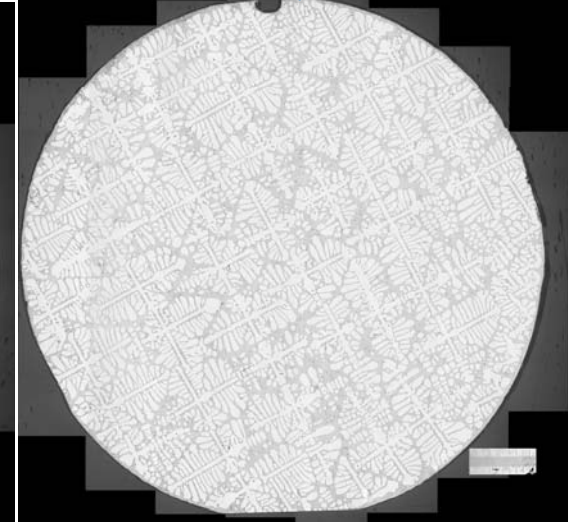


MICAST6: 20 K cm^{-1}

$5 \mu\text{m s}^{-1}$



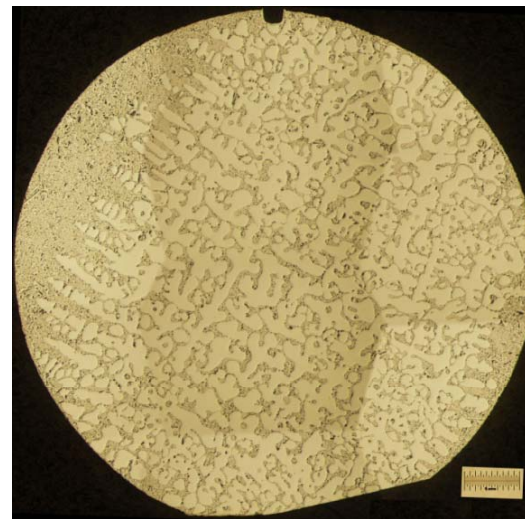
$50 \mu\text{m s}^{-1}$



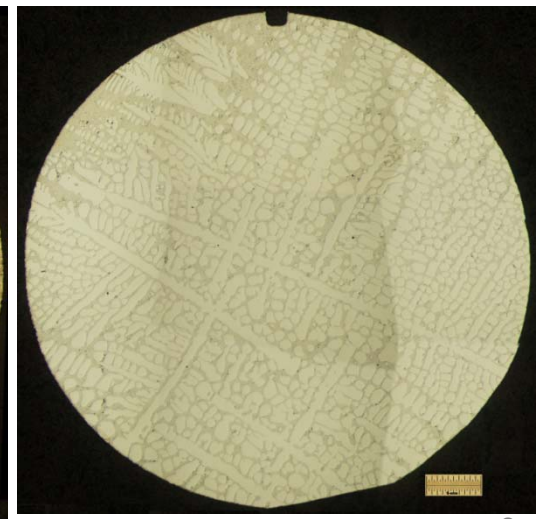
Terrestrial DS:

15 K cm^{-1} →

Convection causes dendrite clustering (steeping) at low thermal gradient and growth speeds during terrestrial DS.

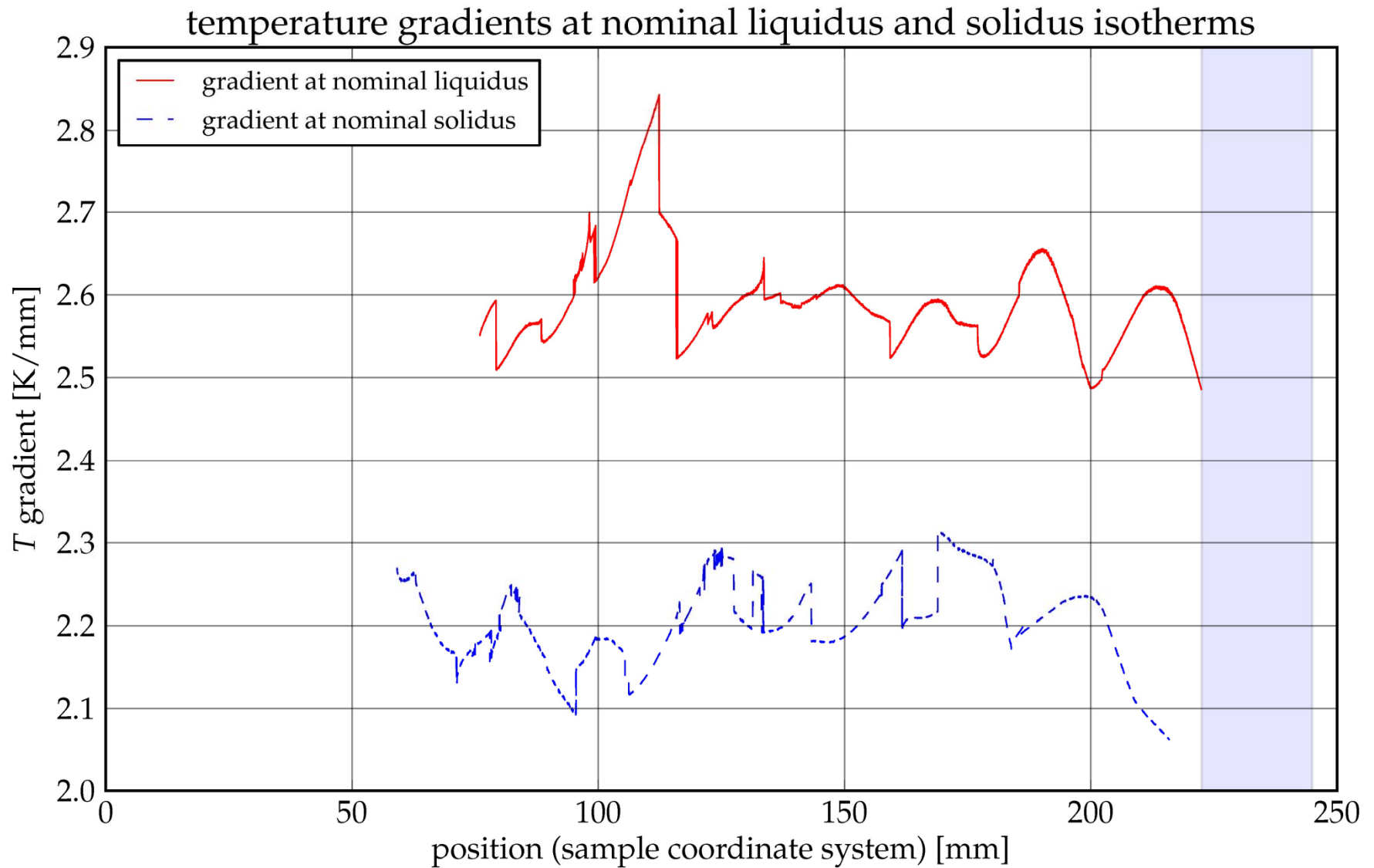


$5 \mu\text{m s}^{-1}$

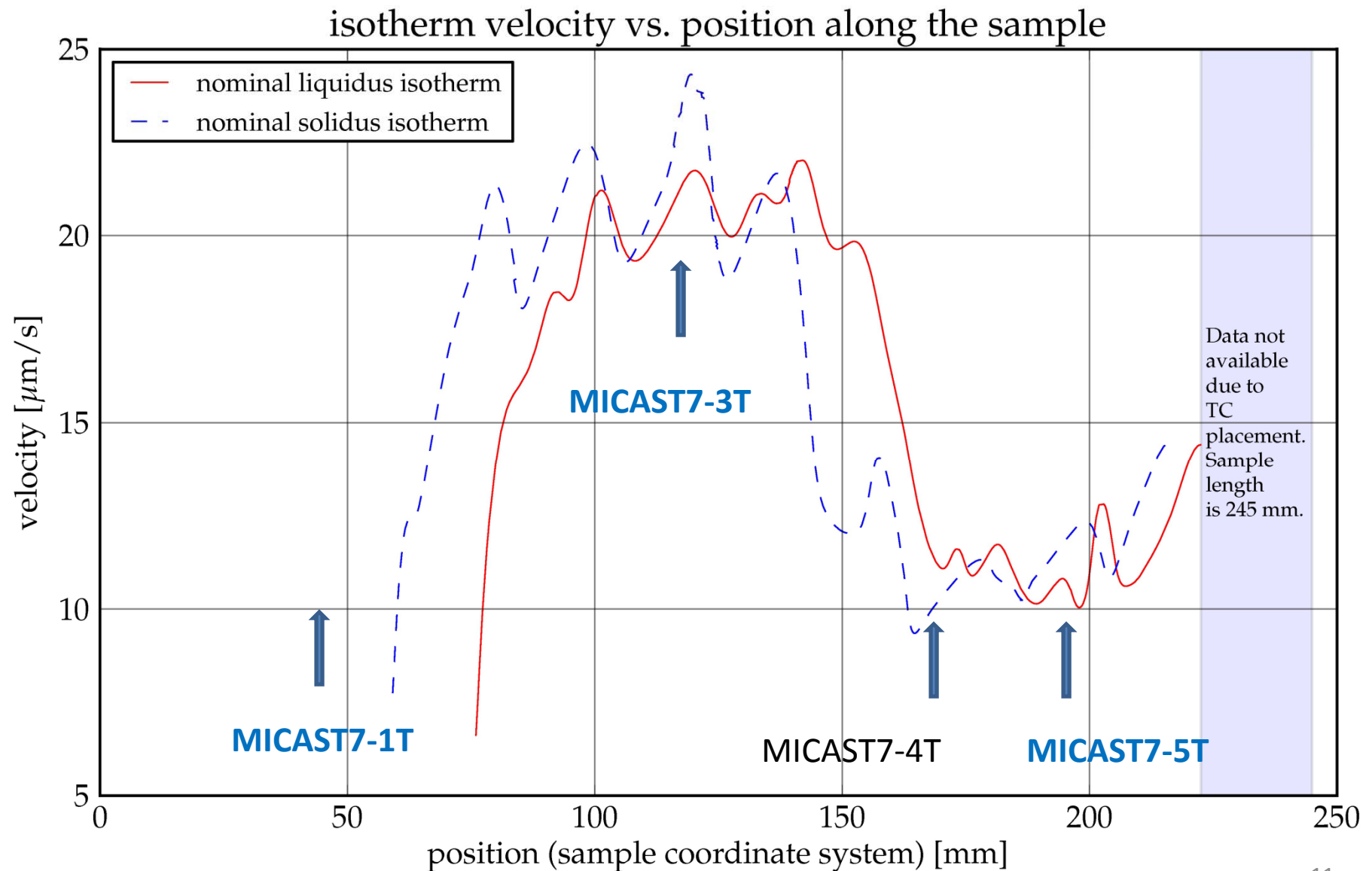


$50 \mu\text{m s}^{-1}$

MICAST7: ESA-SQF (1-hr heat-up, 1-hr hold ($G_l \sim 26 \text{ K cm}^{-1}$): 8.4 cm at $20 \mu\text{m s}^{-1}$, 6.5 cm at $11 \mu\text{m s}^{-1}$



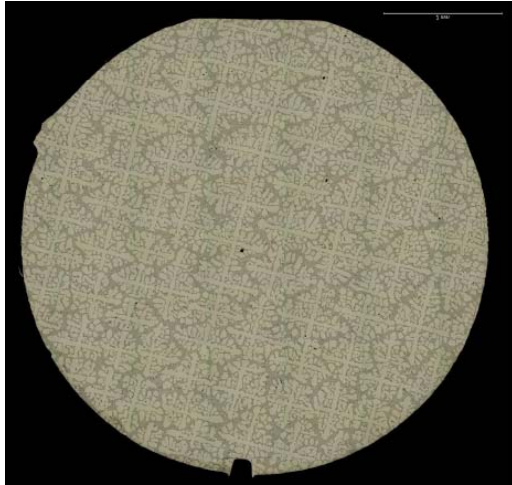
MICAST7: ESA-SQF (1-hr heat-up, 1-hr hold ($G_l \sim 26 \text{ K cm}^{-1}$): 8.4 cm at $20 \mu\text{m s}^{-1}$, 6.5 cm at $11 \mu\text{m s}^{-1}$



Comparison of microstructures: Al-7% Si directionally solidified on ground and on ISS (MICAST7)

MICAST7 SEED

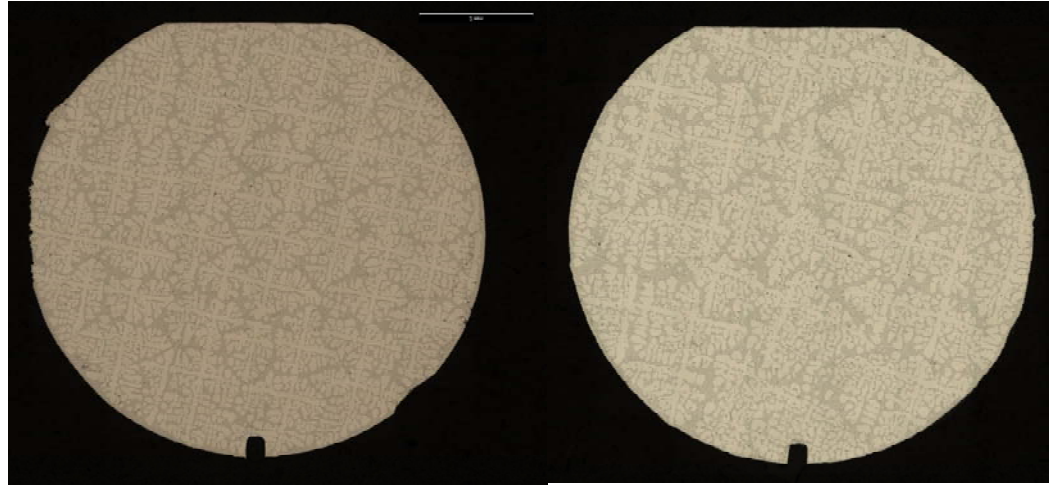
41 K cm⁻¹, 22 μm s⁻¹



MICAST7: 26 K cm⁻¹

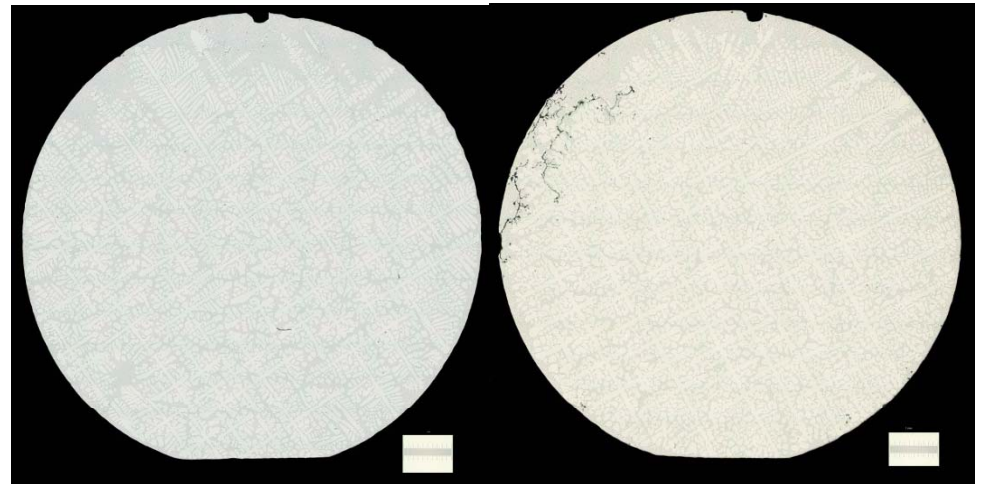
21 μm s⁻¹

11 μm s⁻¹



Terrestrial DS:

24 K cm⁻¹ →



23 μm s⁻¹

10 μm s⁻¹

Growth conditions for MICAST6 and MICAST 7 transverse microstructures examined

Sample ID	G_l , K cm ⁻¹	G_m , K cm ⁻¹	R , $\mu\text{m s}^{-1}$
MICAST6-1	19	18	52
MICAST6-11	20	18.5	47
MICAST6-9	21	19.3	34
MICAST6-7	22.8	20.4	5
MICAST7-3T	26	24	20
MICAST7-4T	26	24	11
MICAST7-5T	26	24	11

Theoretical models : Primary dendrite arm spacing (NEAREST NEIGHBOR SPACING)

$$(m_l G_c^t - G_t) / (4\pi^2 \Gamma T_m / r_t^2) = 1 \text{ for small } R r_t / 2D_l$$

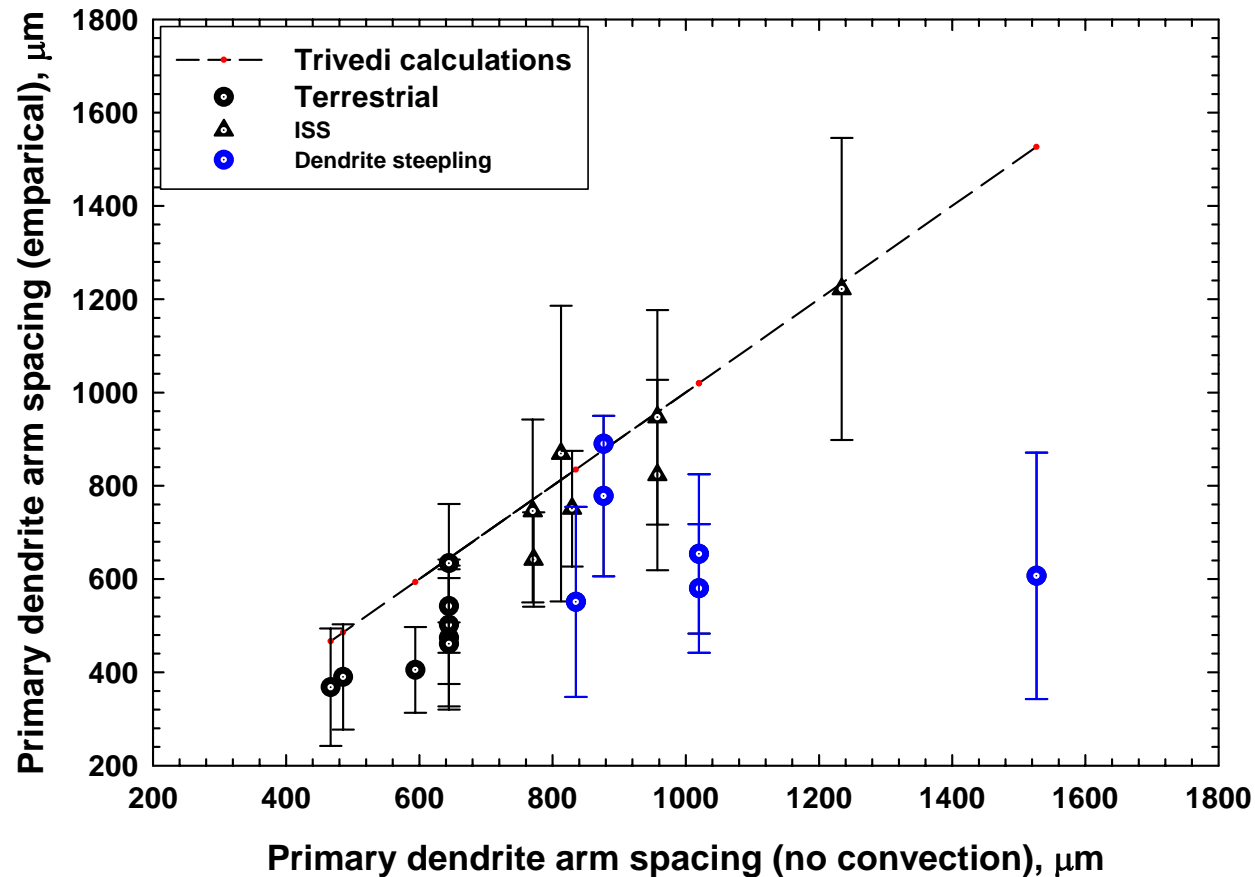
$$r_t = - \frac{G_L \lambda_1^2}{4\sqrt{2} [m_L C_t (1-k) + \frac{D_L G_L}{R}]}$$

Tip radius:	<u>Analytical</u> Trivedi (1980)	<u>Numerical</u> Hunt-Lu (1996)
Primary spacing:	Trivedi (1984)	Hunt- Lu (1996)

Physical Properties for Al- 7 wt% Si

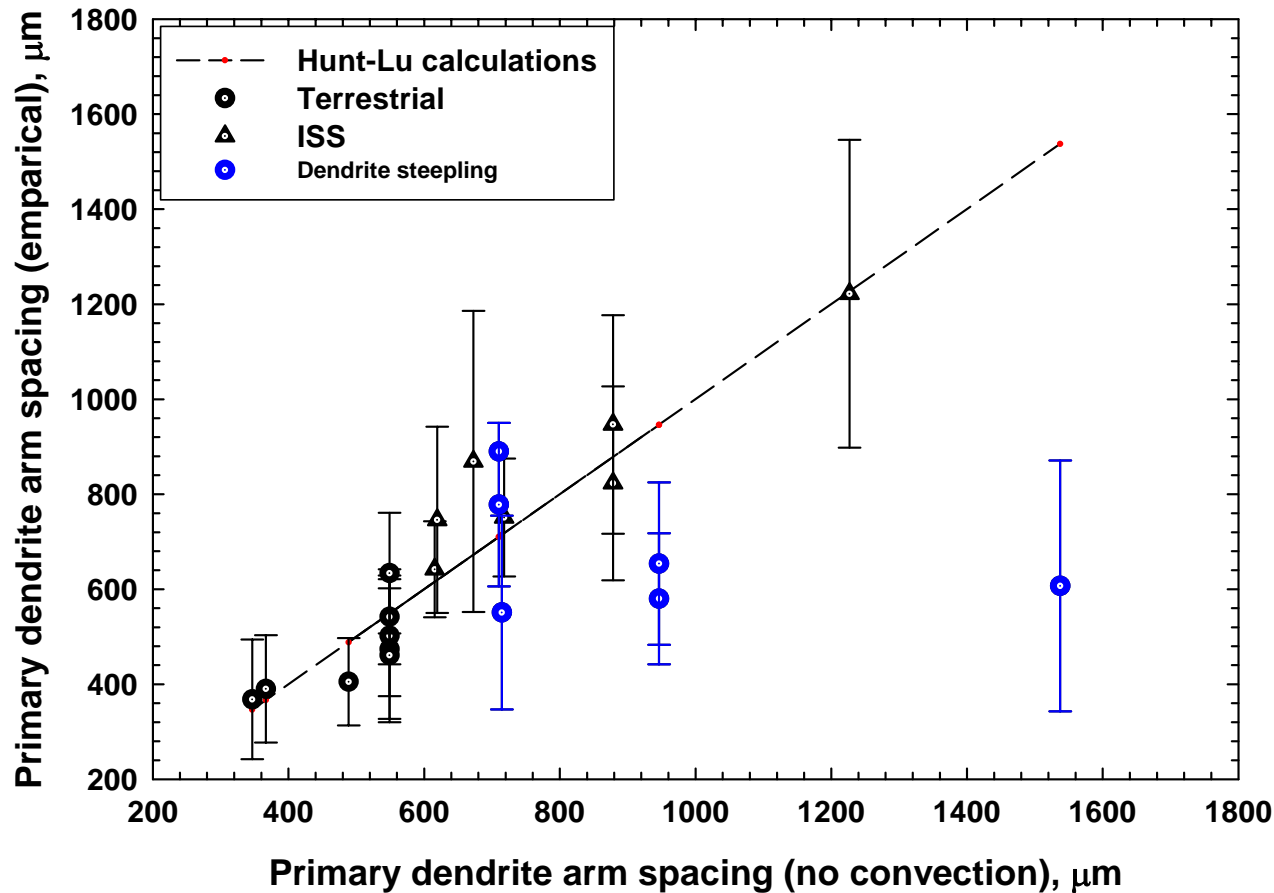
Co	7 wt% Si	
m_l	-6.31 K/ wt% Si	Metals Handbook, vol 8(1973)
k	0.1	
Γ	0.196 $\mu\text{m K}$	Gunduz and Hunt (1985)
D_l	$4.3 \times 10^{-9} \text{ m}^2/\text{s}$	(Poirier compilation)

Primary dendrite arm spacing compared with Trivedi calculations



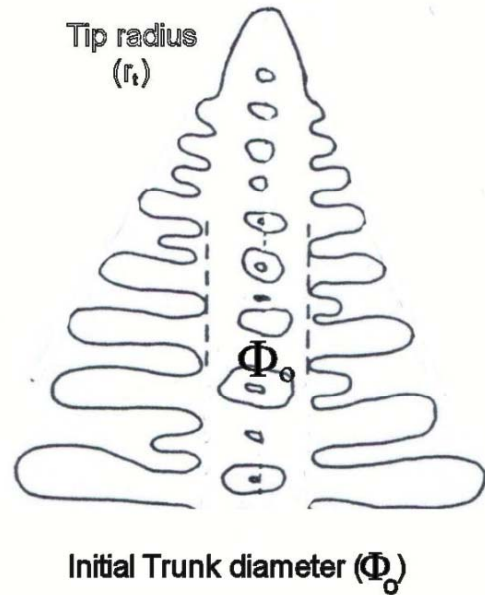
- ISS-DS: Good agreement with predictions from Trivedi model .
- Terrestrial DS ("Not steeped") : Good agreement with predictions from Trivedi model.
- Terrestrial DS ("steeped"): Convection decreases primary dendrite arm spacing.

Primary dendrite arm spacing compared with Hunt-Lu calculations

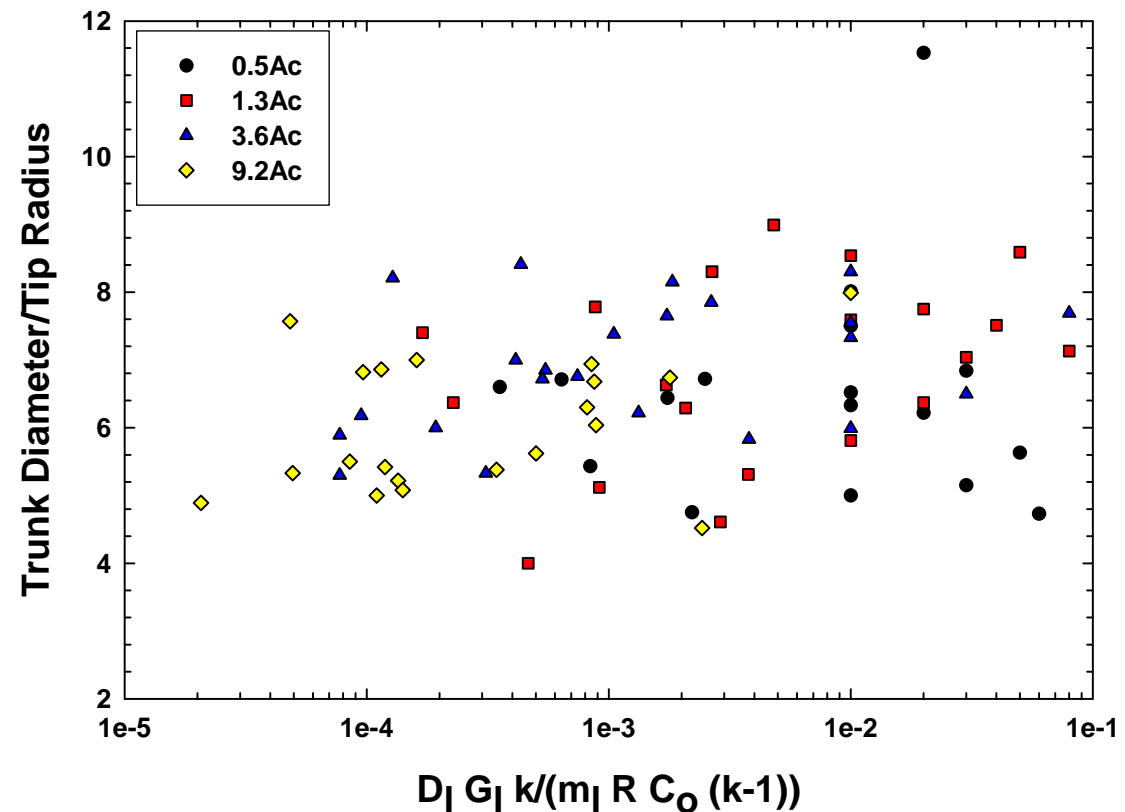


- ISS-DS: Good agreement with predictions from Hunt-Lu model.
- Terrestrial DS ("Not steeped") : Good agreement with predictions from Hunt-Lu model.
- Terrestrial DS ("steeped"): Convection decreases primary dendrite arm spacing.

No theoretical model for Primary dendrite trunk diameter (ϕ)



Esaka Thesis (1986): Trunk diameter increases rapidly near the tip till ~ 10 side-branch formations. He measured this **initial trunk diameter (ϕ_0)**. Eighty DS experiments (four SCN-Acetone alloys grown with various R and G_I)

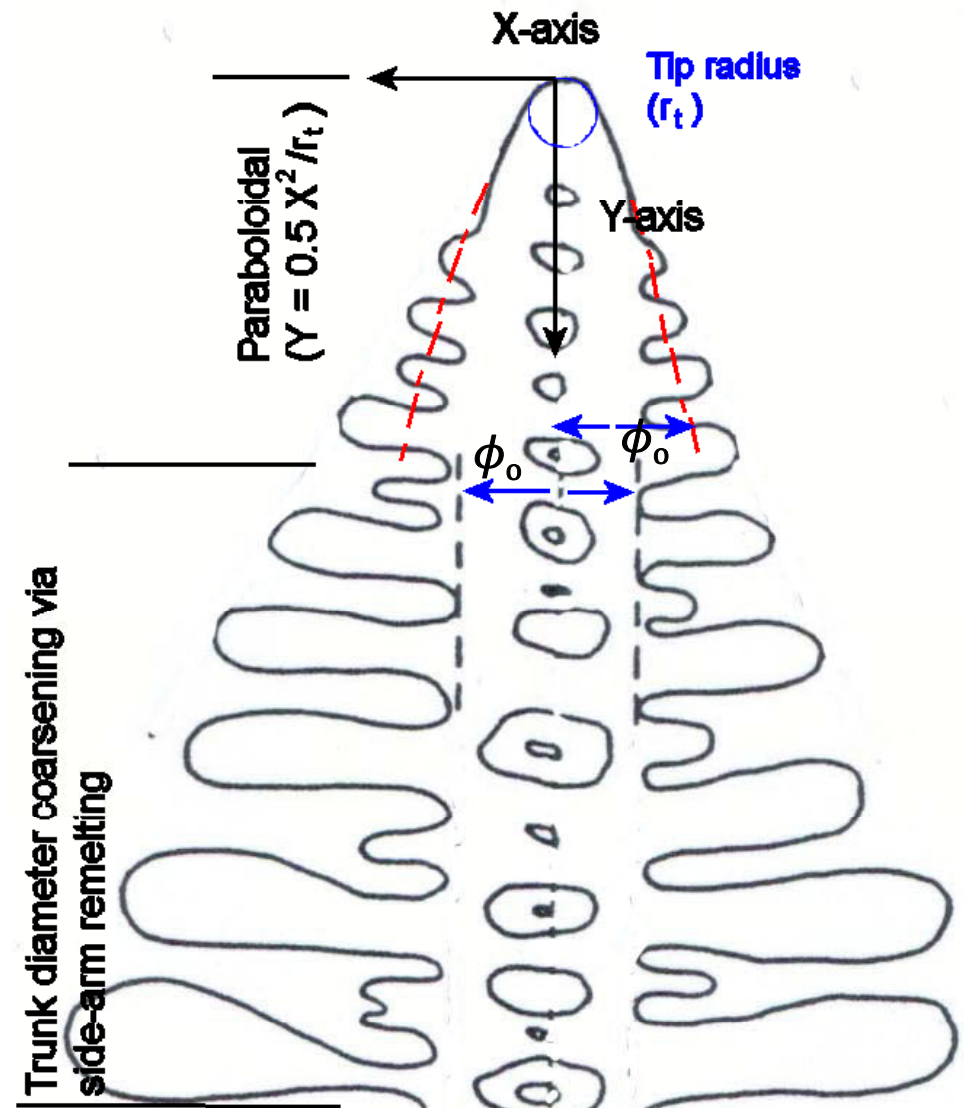


$D_I G_I k/(m_I R C_0 (k-1))$: More branched dendritic morphologies will be located towards the left, and less-branched/cellular towards the right side of the X-axis.

$$(\text{Initial trunk diameter } (\phi_0)/\text{tip radius}) = 6.59 \pm 1.3$$

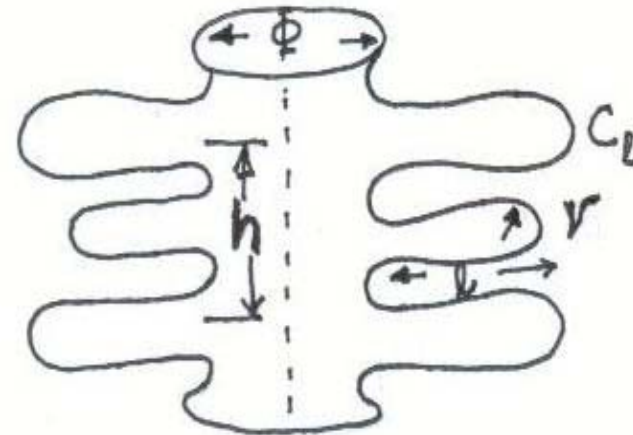
Primary dendrite trunk diameter (ϕ) model

1. The trunk diameter (ϕ) increases rapidly near the tip till time, $t_o = 22 * r_t / R$, when $\phi = \phi_o = 6.59 r_t$ (paraboloidal envelope near tip).



Primary dendrite trunk diameter (ϕ) model

- After t_0 the trunk diameter increases via remelting of 4-side arms (r) and deposition of melted arm material on “trunk surface “over length h ” = ϕ .



Assumptions:

- Kirkwood model (1985) of ripening applies.
- Secondary arm melts back because of its curvature.
- Mass of the melted arm deposits on trunk surface where there is negative curvature.

$$\frac{dl}{dt} = \frac{4 D_l \Gamma}{m_l C_l (1-k) r^2} \quad (1)$$

$$\pi \phi h \frac{d\phi}{2 dt} = 4 * \pi r^2 \frac{dl}{dt} \quad (2)$$

$$C_l = C_o + R G_m t / m_l \quad (3)$$

$$\phi^2 \frac{d\phi}{dt} = 32 \frac{D_l \Gamma}{m_l (1-k) (C_o + \frac{R G_m t}{m_l})} \quad (4)$$

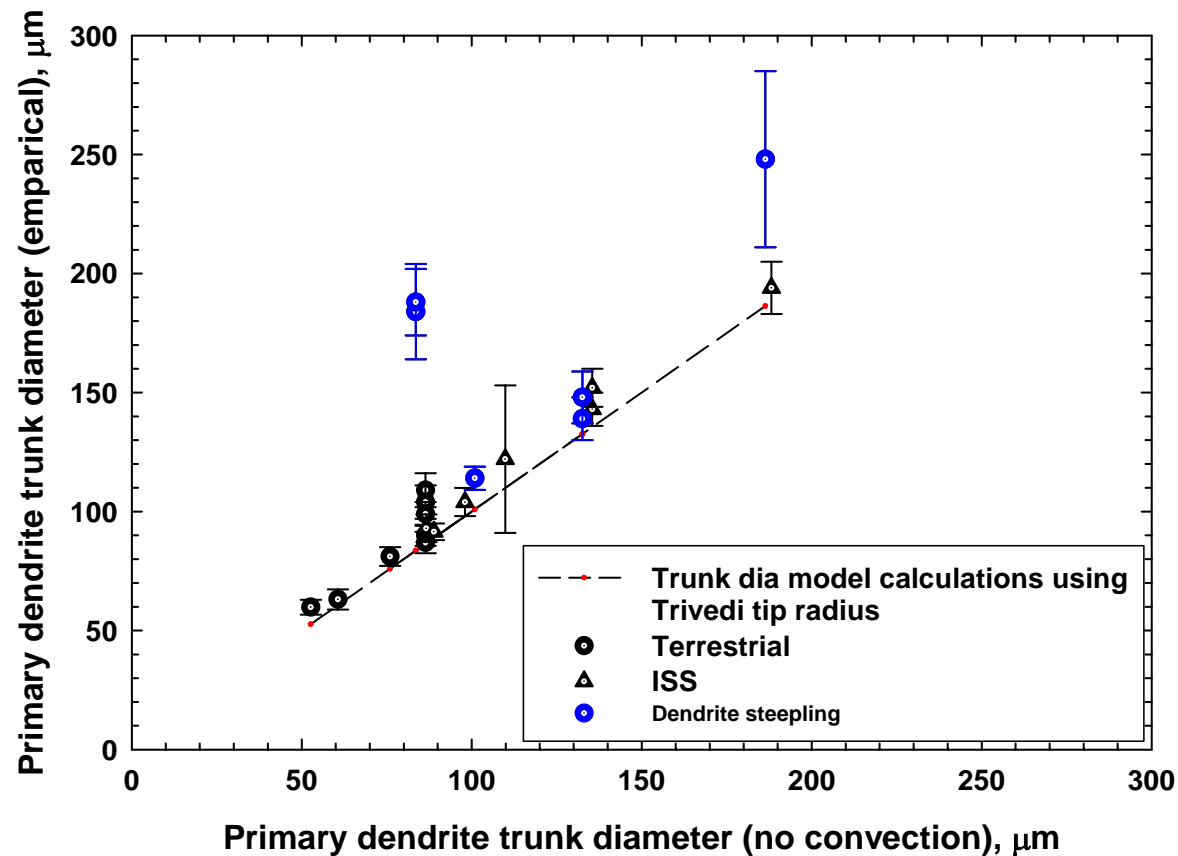
Primary dendrite trunk diameter (ϕ) model

$$\phi^3 = 96 \frac{D_l \Gamma}{R G m (1 - k)} \ln \left\{ \frac{\left(1 + \frac{R G m t}{m_l C_o} \right)}{\left(1 + \frac{R G m t_o}{m_l C_o} \right)} \right\} + \Phi_o^3$$

Mushy zone freezing time $\sim m_l(C_E - C_o)/R G_m$

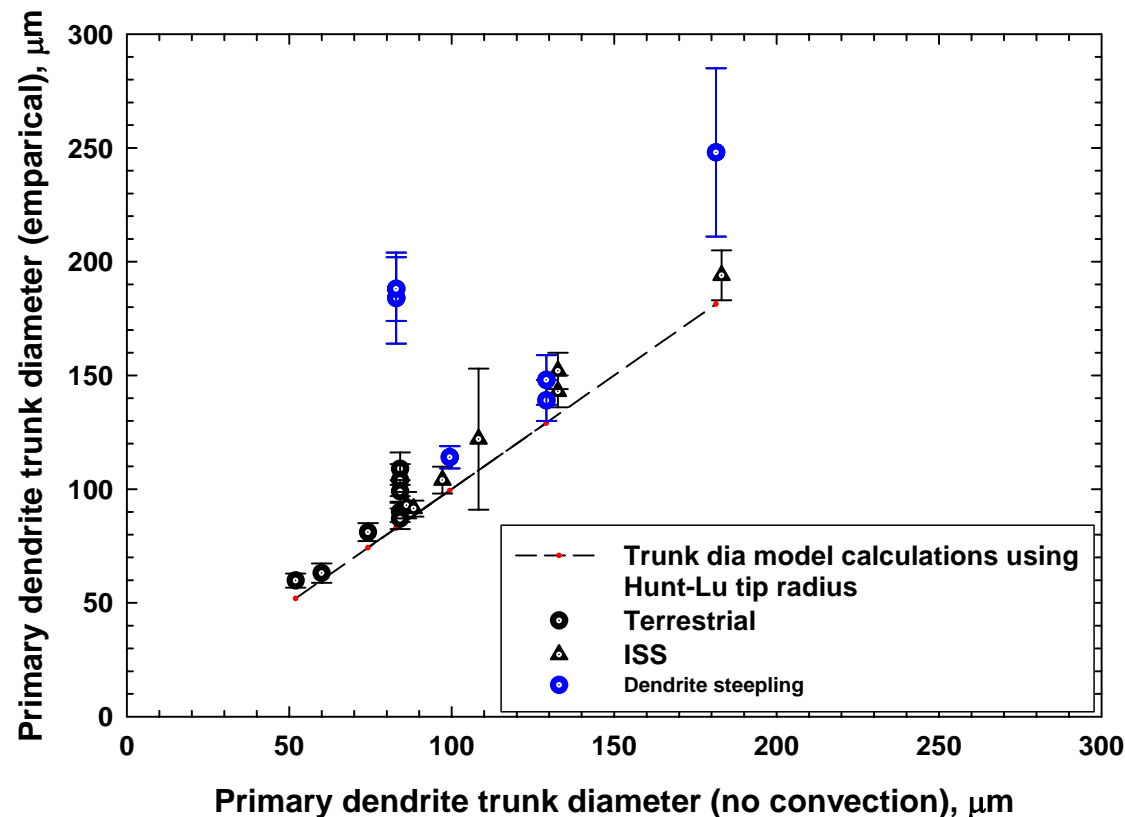
Use tip radius (r_t) predicted from Trivedi (1980) or Hunt-Lu (1996) models to get the initial trunk diameter $\phi_o = 6.59 r_t$ in order to predict the processing parameter dependence of “Primary dendrite trunk diameter” from above relationship.

Primary dendrite trunk diameter as compared to trunk diameter model calculations, using r_t (Trivedi)



- ISS-DS: Good agreement with predictions from the trunk-diameter model.
- Terrestrial DS ("Not steeped") : Good agreement with predictions from model.
- Terrestrial DS ("steeped"): Convection increases trunk diameter.

Primary dendrite trunk diameter as compared to trunk diameter model calculations, using r_t (Hunt-Lu)



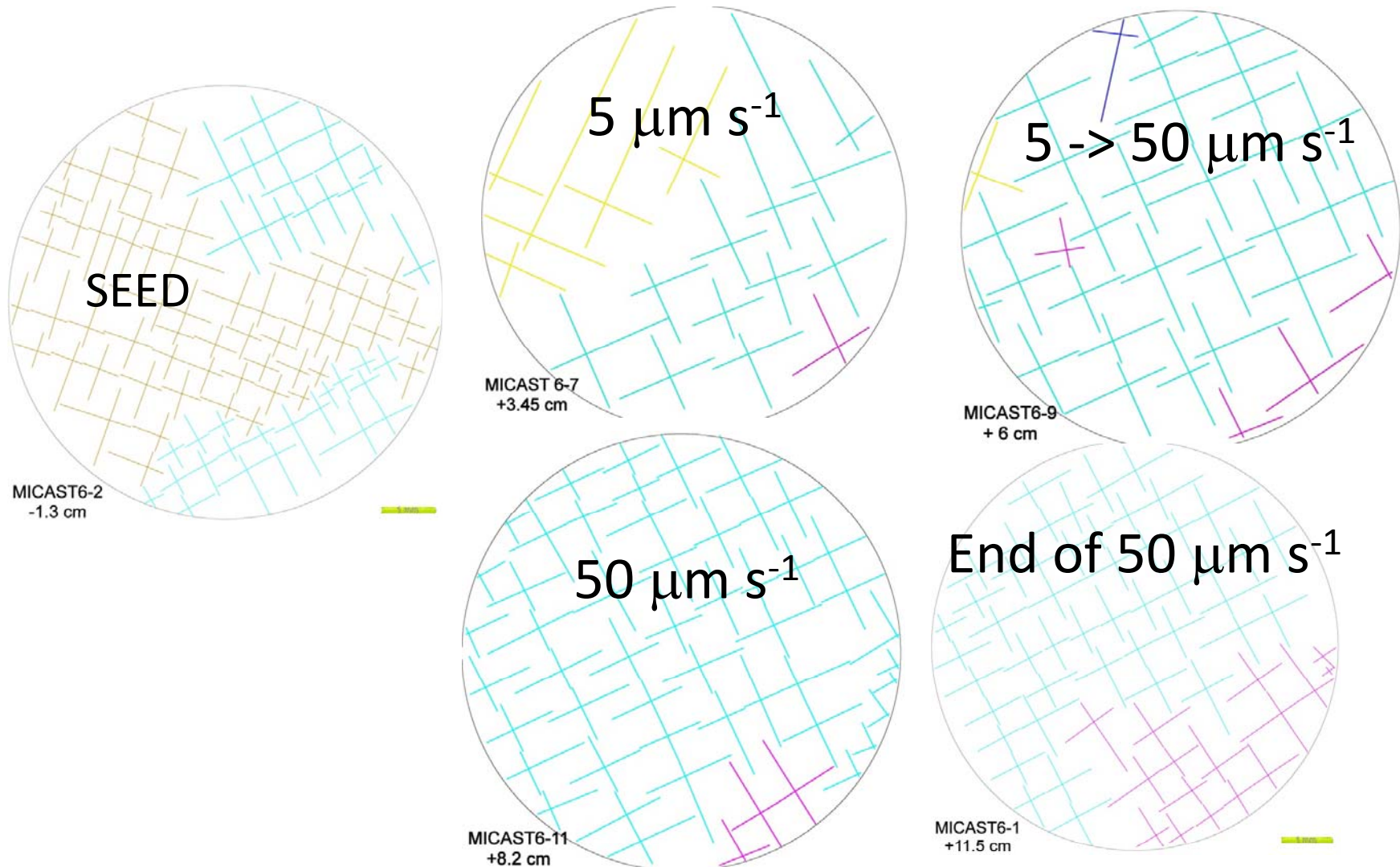
- ISS-DS: Good agreement with predictions from the trunk-diameter model.
- Terrestrial DS (“Not steepled”) : Good agreement with predictions from model.
- Terrestrial DS (“steepled”): Convection increases trunk diameter.

ISS samples show better agreement with calculations from the models than terrestrial samples (primary spacing and trunk diameter (Observed/Predicted))

	Trivedi		
	ISS-samples	Terrestrial (no steeping)	Terrestrial (steeping)
Primary dendrite arm spacing/calculated from model	0.945± 0.0833 ($\sigma/\mu=0.09$)	0.791± 0.0931 ($\sigma/\mu= 0.12$)	0.695± 0.223 ($\sigma/\mu=0.32$)
Primary dendrite trunk diameter/calculated from model	1.069± 0.0361 ($\sigma/\mu= 0.05$)	1.113± 0.0890 ($\sigma/\mu=0.08$)	1.513± 0.560 ($\sigma/\mu=0.37$)

SURPRISES??

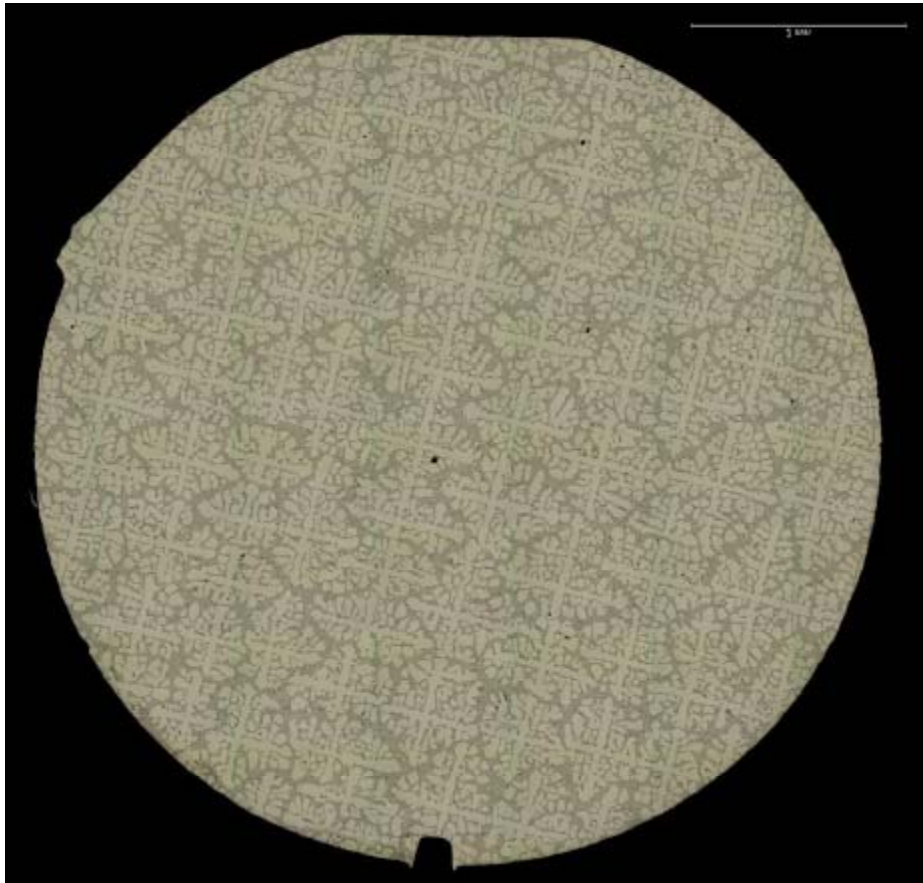
MICAST-6: Grains elimination and formation along DS length



SPURIOUS GRAINS DURING DS IN THE ABSENCE OF CONVECTION??

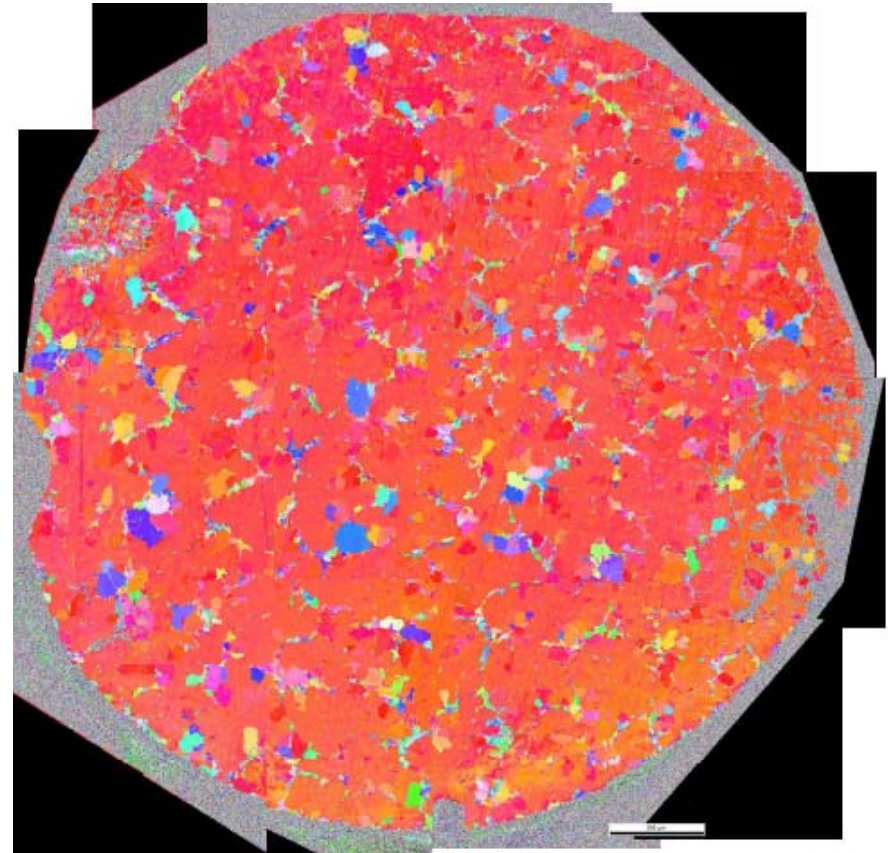
MICAST7-1T (Ground processed seed crystal)

Al – 7wt. % Si, $V = 22 \mu\text{m s}^{-1}$, $G_I = 41 \text{ K cm}^{-1}$

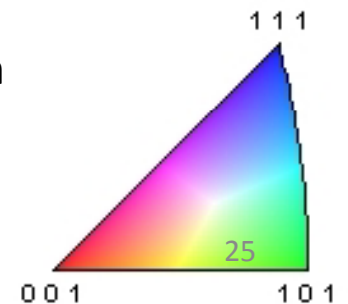


Transverse optical microstructure

Very good [100] alignment, some spurious grains/arms

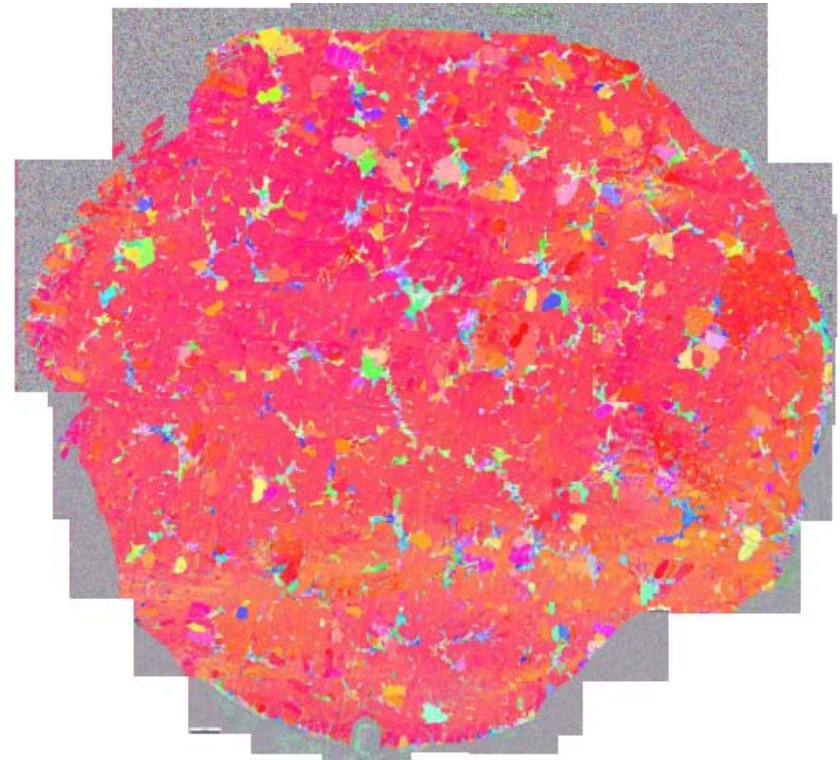
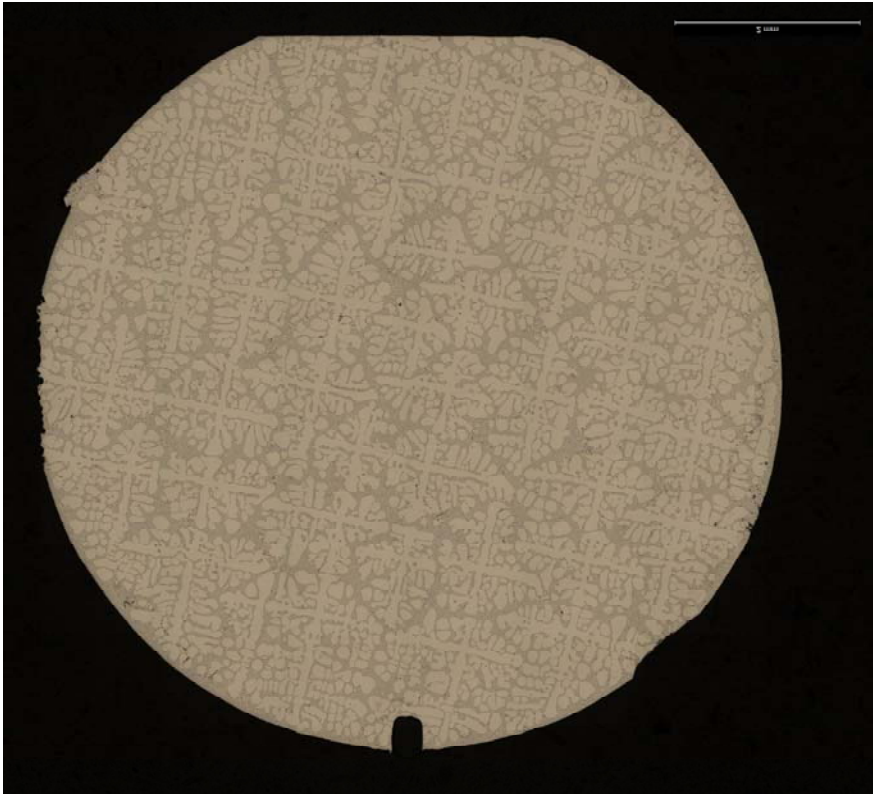


Composite EBSD Scan

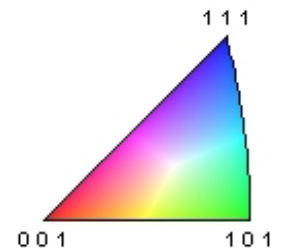


Microgravity Processed MICAST 7

MI CAST7 – 3T ($20 \mu\text{m s}^{-1}$, $G_I = 26 \text{ K cm}^{-1}$)

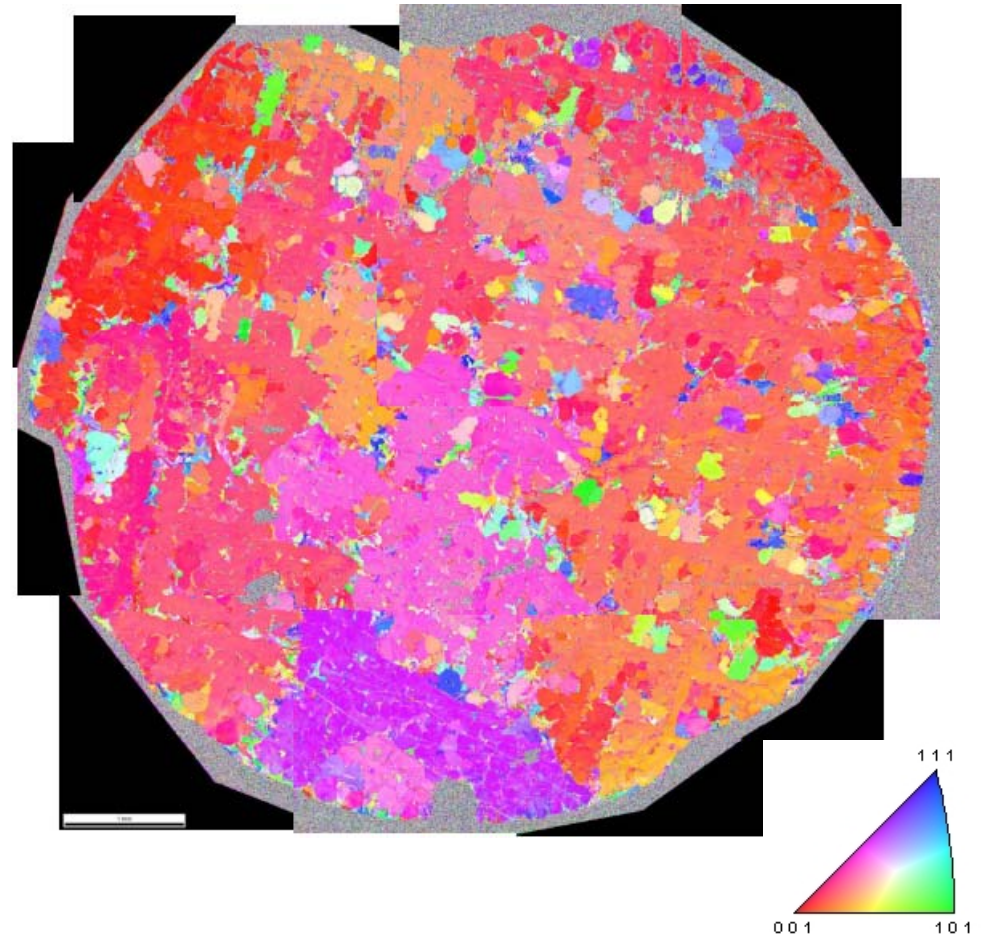
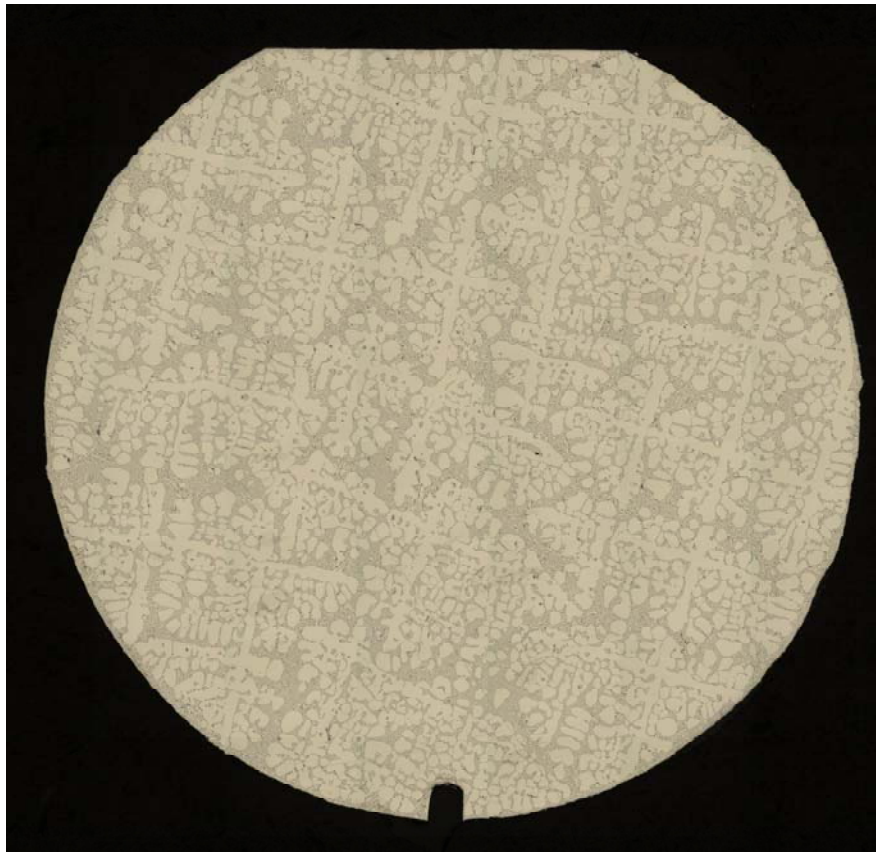


Very good (better) alignment, less spurious grains



Microgravity Processed MICAST 7

MICAST7 – 5T ($10 \mu\text{m s}^{-1}$, $G = 28 \text{ K cm}^{-1}$)



Very poor alignment, very many spurious grains

Conclusions

- Primary dendrite arm spacings of Al-7 wt% Si alloy directionally solidified in low gravity environment of space (MICAST-6 and MICAST-7: Thermal gradient ~ 19 to 26 K cm^{-1} , Growth speeds varying from 5 to $50 \mu\text{m s}^{-1}$) show a good agreement with predictions from Hunt-Lu and Trivedi models.
- Primary dendrite trunk diameters of the ISS processed samples show a good fit with a simple analytical model based on Kirkwood's approach, proposed here.
- Natural convection,
 - decreases primary dendrite arm spacing.
 - appears to increase primary dendrite trunk diameter.
- Spurious grains formed during DS in “low- $g < 10^{-4} \text{ g}$ ”??

Acknowledgements

- NASA
- ESA
- DLR-MUSC
- ALCOA